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SCHOTT Low Temperature Bonding for Precision Optics

Carol Click, Leo Gilroy and Dave Vanderpool
SCHOTT North America

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Outline of topics to be covered

- Overview of Low Temperature Bonding
  - Process overview
  - Materials that can be bonded
- Measured properties of LTB components
  - Bond thickness
  - Mechanical strength
  - Total change in length
  - Stability
  - Stress birefringence
- Summary and future work

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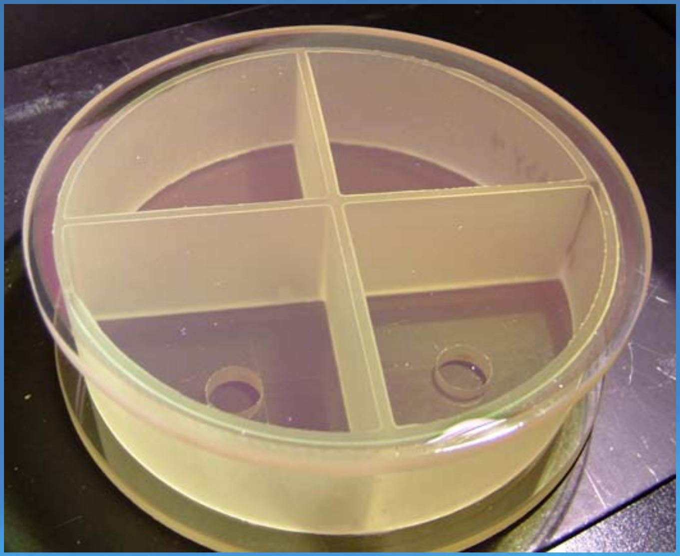
Overview of LTB



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What is LTB?

- Low temperature (<120°C)
  - Fabrication is done at room temperature and cured at 120°C
- Aqueous and inorganic
  - No organic out-gassing issues
- Permanent
  - High aqueous durability and is stable from 30K to 1000K
- Optically transparent
  - More transparent than the Zerodur®
- Mechanically robust
  - Bonded components have strengths similar to monolithic components
- Vacuum compatible
  - Bonded components are ultra-high vacuum compatible and survive coating chambers
- Post-bond process-able
  - Bonded components can be polished after curing without degradation of the bond

SCHOTT Proprietary LTB Process

- Components are prepared for bonding
  - Grinding and polishing of faces that will be bonded
  - Aqueous based cleaning to remove surface contamination and activate the surface to a hydrophilic state
- Solution is applied to bonding faces and the components are brought into contact
- Alignment of the component is performed in the first 2 minutes
- Components are allowed to sit undisturbed for 6-12 hours
- Component is cured for ~1 week – depending on sample geometry
- Component is now ready for post-processing
  - Polishing
  - Coating
- Small voids are cosmetic and do not appear to affect overall quality of the bond

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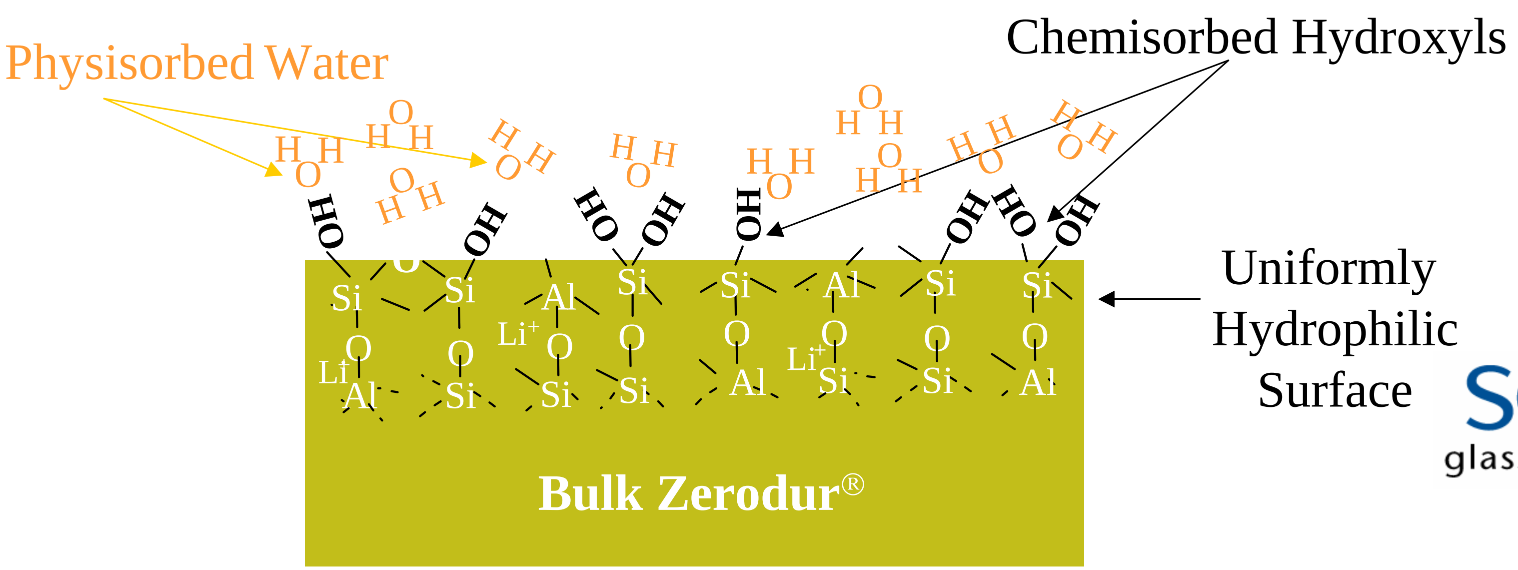
Surface activation for LTB

- Surface must be hydrophilic for uniform wetting of the surface and resulting uniform bonds
  - Hydrophilic vs. hydrophobic is measured by the contact angle
  - Small contact angles indicate a hydrophilic surface which is desirable for bonding (typically <30º)
- Hydrophilic surfaces have both chemically absorbed hydroxyl ions and physically absorbed water molecules
  - The more uniform the coverage of the water molecules and hydroxyl ions, the better the overall bond quality



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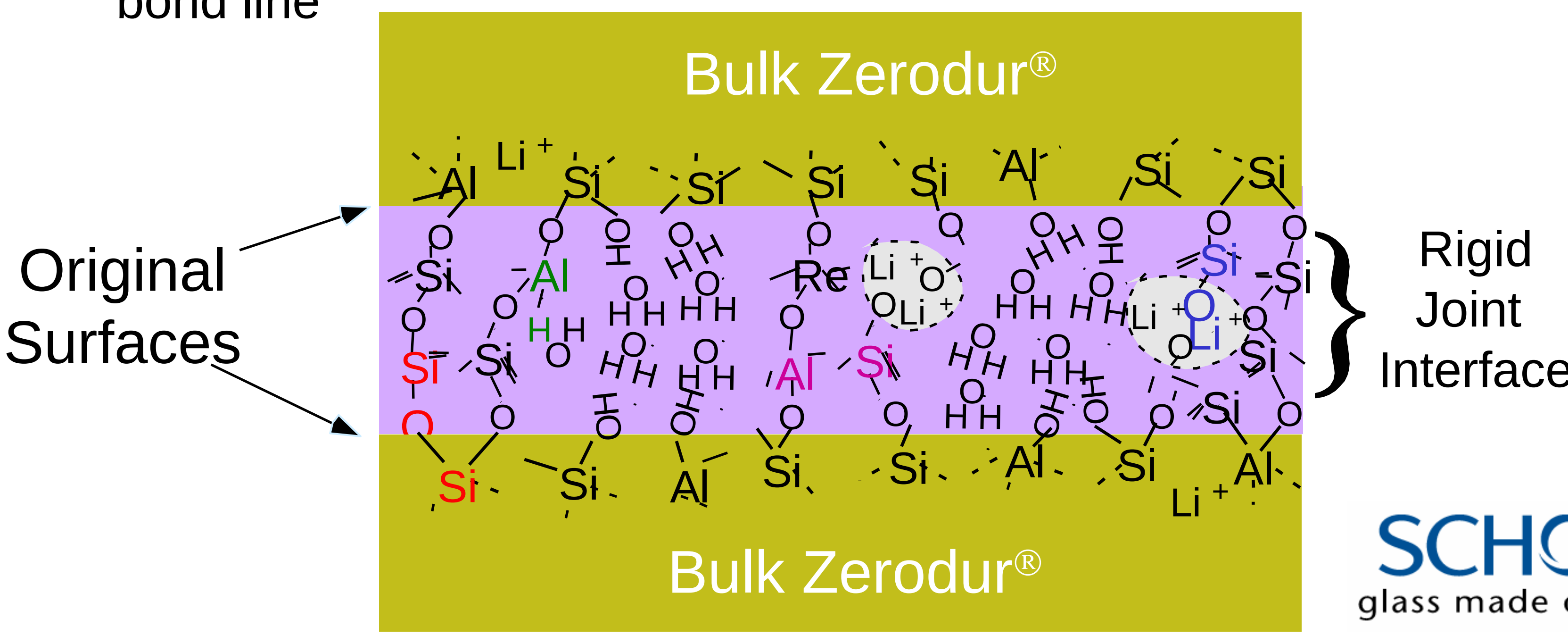
Reactive bonding is initiated by the inorganic bonding solution being applied to the joint

- Multiple bonding mechanisms are present:
  - Hydrogen bonding
  - Alkali ion to non-bridging oxygen bonding
  - Si – O – Si network bonding
- Joint cures rigid while dehydration of the joint occurs through the bond line



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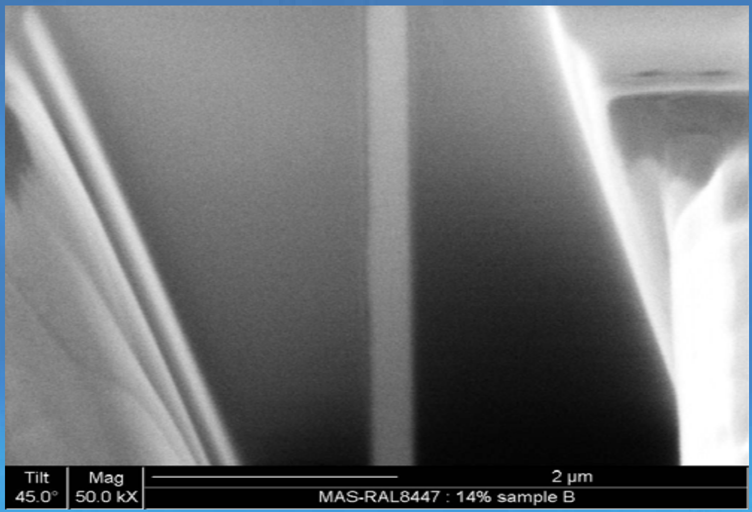
Post-processing

- After bonding, the components are cured
  - From room temperature to 120^{o}C
- After curing, the components can be thermal cycled (77K to 200^{o}C) to remove residual stress
- After curing and thermal cycling the joints are stable and are capable of being polished and coated into a final component

LTB compatible materials

- Glasses – Silicates, phosphates, multi-component glasses, heavy metal oxides
- Glass ceramics – **Zerodur®** and Ceran®
- Crystalline materials – Sapphire, silicon, GaAs, Nd-YAG, Indium Tin Oxide, $LiNbO_3$, CaF_2, MgF_2
- Metals – Invar, copper/tungsten, aluminum, copper

Measured properties of LTB bonded components



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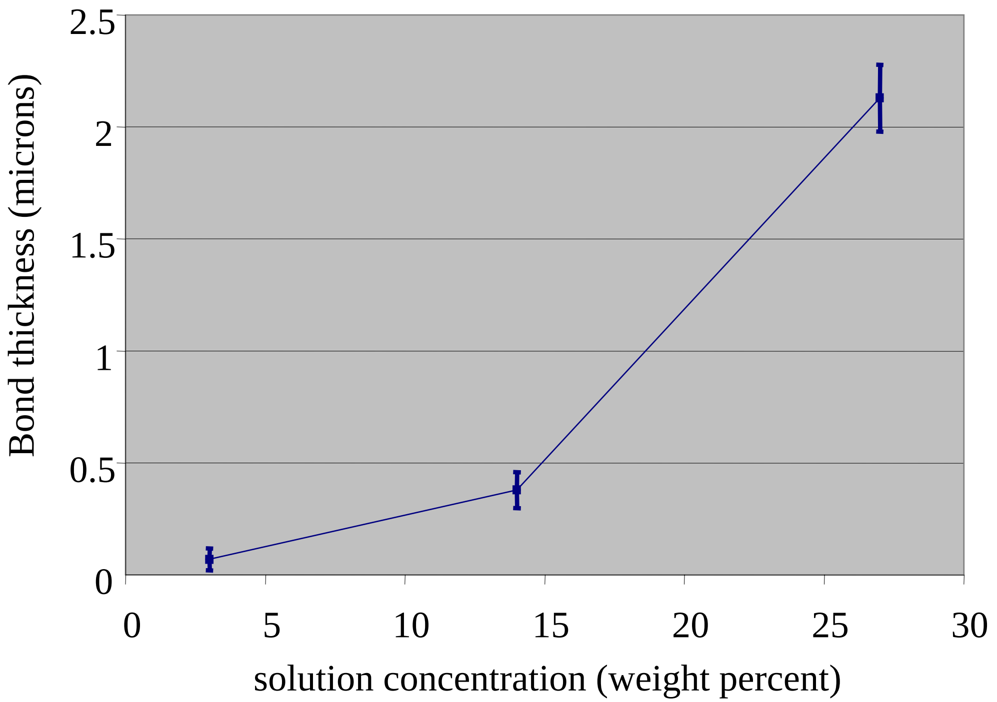
Bond thickness is dependent upon solution chemistry

- The bond thickness has been measured using a Focused Ion Beam on a Scanning Electron Microscope (FIB/SEM)
- The bond thickness can be varied between 100nm and 2.2μm on λ/3 flat Zerodur® surfaces



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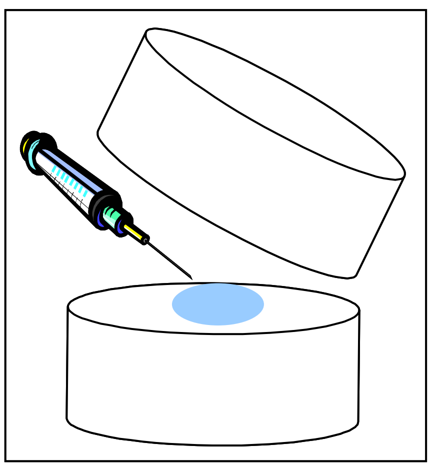
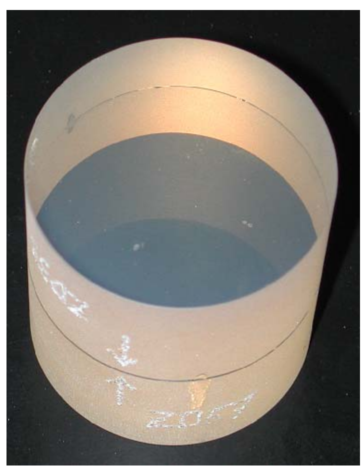
Strength testing of bonded Zerodur® assemblies

- A silicate-based LTB solution was prepared and 2” diameter, 2” tall samples, with either polished or fine ground surfaces, were bonded together (“hockey-pucks”)
- Bonded samples were either cured at room temperature or at a maximum temperature of 110 °C (Standard Cure)
- Rods were core-drilled and 4-pt flexure testing was done in accordance with ASTM 1161-02C using circular cross sections
- Core drilling impart stress and flaws to the surface, so this data gives us a rough order of measure on strength values!

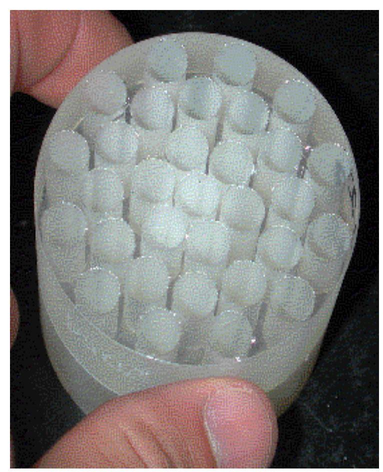
1. Bond



2. Cure

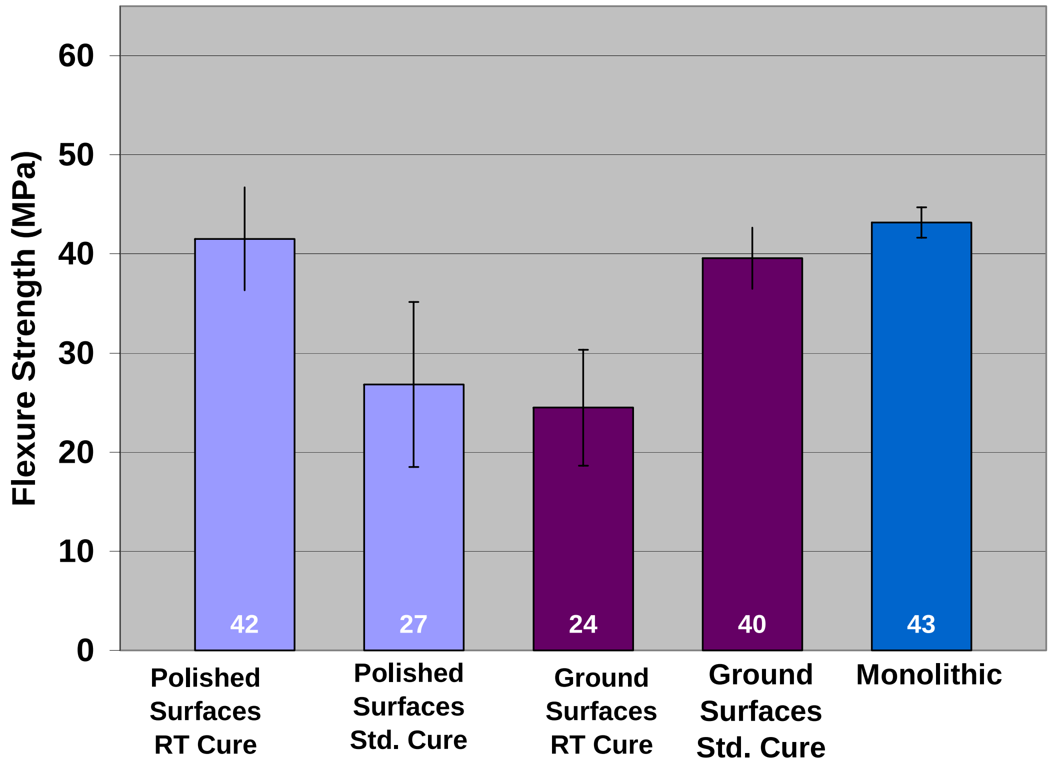


3. Core Drill



4. Test Strength

Bonded components can be produced with similar strengths to monolithic Zerodur®



- Assemblies made using polished surfaces ($\lambda/3$) are stronger with a room temperature cure as opposed to a high temperature treatment
- Assemblies made with ground surfaces should be heat treated to provide strength values that approach that of the monolithic

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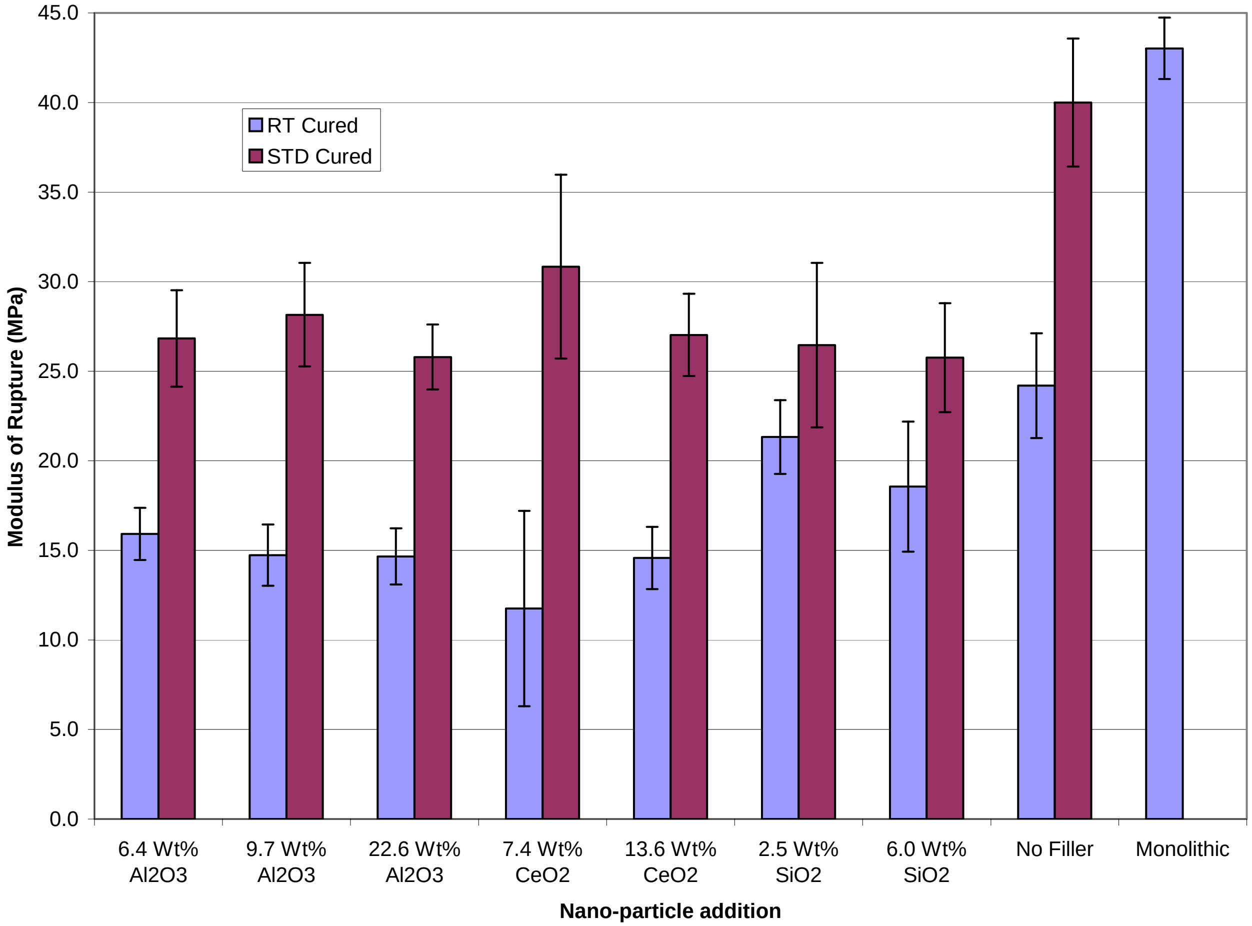
Nano-particles were added to increase the strength of fine ground bonded Zerodur®



- Nano-particle additions did not increased the strength of the bonded components, which would have indicated successful gap filling

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MOR of core drilled Zerodur® can be altered via surface treatment

- Monolithic Zerodur® was core drilled into 5mm diameter rods (70mm in length)
- The modulus of rupture was measured on three sets of samples

Core Drilled Monolithic Zerodur	Modulus of Rupture (MPa)	Weibull Modulus
As received	59.3 +/- 5.5	11.2
Dip coated in bonding solution	51.7 +/- 6.7	8.0
Acid etched	126.3 +/- 36.8	3.4

- Acid etching greatly increases the strength of monolithic Zerodur®, and it also increases the distribution of flaw sizes on the surface
- Dip coating in bonding solution does not have the desired effect of “flaw healing” on the surface of the rods

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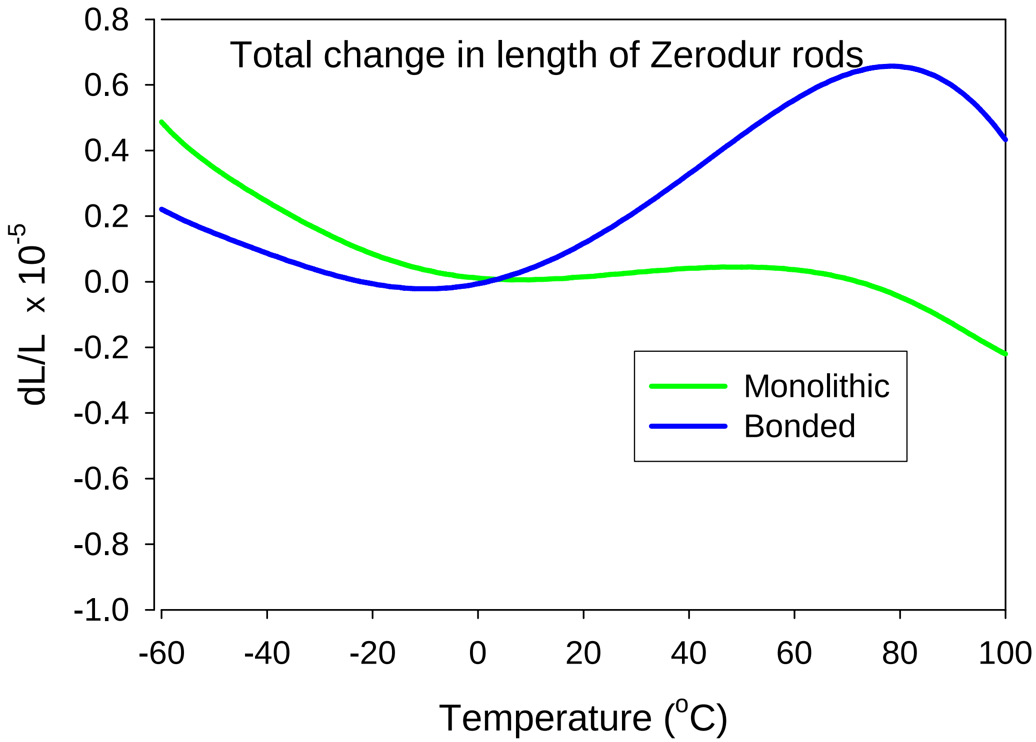
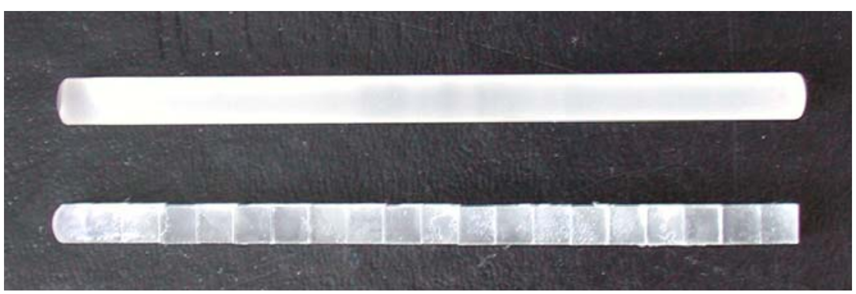
Total change in length (TCL) of Zerodur® rods

- 20 disks of Zerodur® were bonded together to form a rod
- Rod contained a maximum of ~0.02 vol% of bonding material after curing
- Rods are 100 mm in length, 5mm in diameter





- TCL (-60 – 100ºC)
  - Monolithic – 5.7 ppm
  - Bonded – 6.8 ppm

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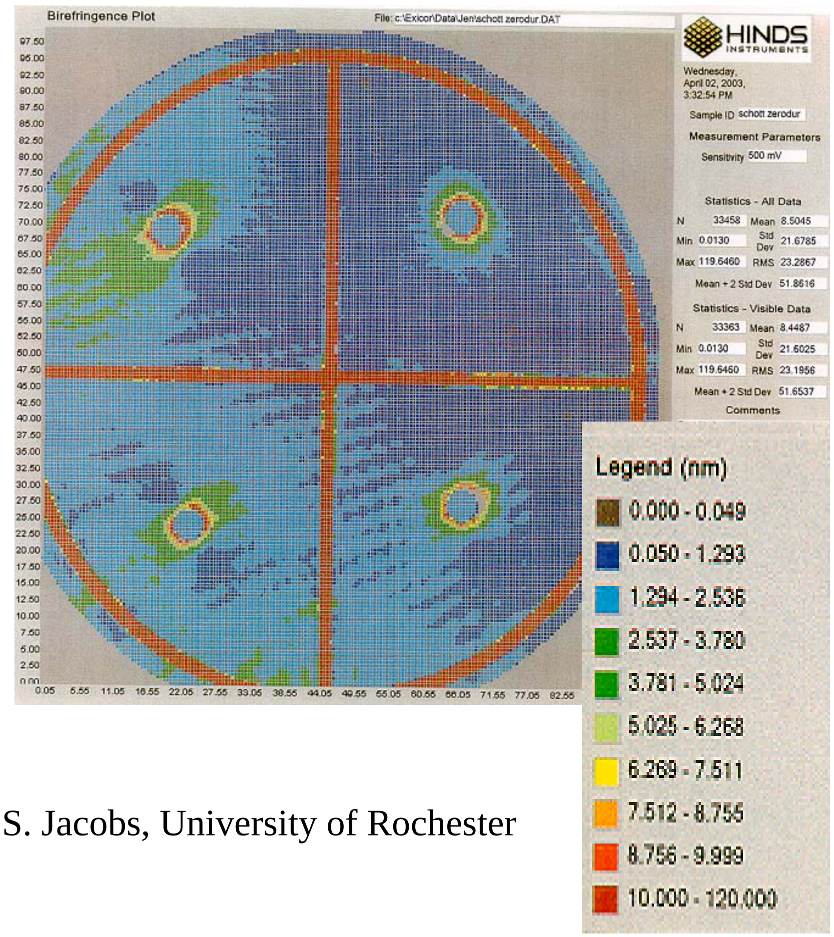
Bonded components are vacuum stable

- A bonded Zerodur® valve has tested to 10^{-12} mbar/liter/sec (ultra-high vacuum range)
  - Leak rate below current detection limit (Calibrated helium leak, 2.6×10^{-8} std. cc/sec); lower than that typically specified by manufacturers of UHV valves

- Bonded Zerodur® light-weight mirror assemblies have survived aggressive coating chambers (CVD) (high temperature and pressure fluctuations)
  - Both fine ground cores and polished cores survived this coating procedure with no significant change in surface figure after coating

Bonded Zerodur® components have low stress birefringence

- Stress birefringence was measured on bonded pieces that had water jet cut cores
  - ➢ The maximum retardance was 40 nm/cm without acid etching
    - ➢ This is an unacceptable level due to the amount of residual stress it indicates
  - ➢ The maximum retardance was reduced to 8 nm/cm with acid etching
    - ✓ This is an acceptable level within specification for fine annealed optical glass



S. Jacobs, University of Rochester

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Summary and future work

Summary of SCHOTT LTB

- A flexible low temperature bonding solution
- Can be used to build complex structures
- Can be polished and coated after bonding
- Thin bond lines for low distortion
- Near monolithic strength for bonded components

Future work

- Measure the thermal expansion of bonded Zerodur® components in a very controlled and precise system, as to measure the CTE of the joint material
- Determine the shrinkage characteristics of LTB bonds
- Determine post processing steps necessary to obtain surface figure stability of $\lambda/50$

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